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कॉस्मेटिक उद्योग के  
लिए तिल का तेल — विशिष्टि  
( पहला पुनरीक्षण )

Sesame Oil for  
Cosmetic Industry — Specification  
( First Revision )

ICS 71.100.70

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## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1985. The first revision was taken up to keep pace with the latest technological developments. In this revision, limiting requirement of hexane has been incorporated and the formula for determination of peroxide value (**B-3**) has been modified. Further, amendment No. 1 (July 2004) to the previous version has been incorporated in this revision. An optional requirement of total aflatoxin has been added (25 ppb, max ) and is determined using High Performance Liquid Chromatography (HPLC)

Both the types of oil, namely expressed and solvent extracted are covered in the present standard. All other changes considered necessary to align the standard with others in the series for raw materials and finished cosmetics have been included.

The composition of the Committee responsible for formulation of this standard is given at Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# Indian Standard

## SESAME OIL FOR COSMETIC INDUSTRY — SPECIFICATION

( First Revision )

### 1 SCOPE

**1.1** This standard prescribes requirements and methods of sampling and test for sesame oil for cosmetic industry.

**1.2** For sesame oil for edible purposes and for manufacture of *VANASPATHI* and refined oil a separate standard, IS 547-‘Sesame oil — Specification’ has been published.

### 2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below.

<i>IS No.</i>	<i>Title</i>
548 (Part 1) : 1964	Methods of sampling and test for oils and fats: Part 1 Sampling, physical and chemical tests ( <i>revised</i> )
548 (Part 2) : 1976	Methods of sampling and test for oils and fats: Part 2 Purity test ( <i>third revision</i> )
1448 (Part 21) : 2019/ ISO 2719	Methods of test for petroleum and its products: Part 21 Determination of flash point — Pensky-Martens closed cup method ( <i>third revision</i> )
3470 : 2017	Hexane, food grade — Specification ( <i>second revision</i> )
IS 16287 : 2015/ ISO 16050 : 2003	Foodstuffs — Determination of aflatoxin B <sub>1</sub> , and the total content of aflatoxins B <sub>1</sub> , B <sub>2</sub> , G <sub>1</sub> and G <sub>2</sub> in cereals, nuts and derived products

### 3 TYPES

The material shall be of two types:

- a) *Type 1* — Refined oil; and
- b) *Type 2* — Raw expeller oil.

### 4 REQUIREMENTS

#### 4.1 Description

**4.1.1 *Type 1*** — The material shall be sesame oil obtained from clean and sound sesame seeds (*Sesamum indicum* Linn., syn. *Sesamum orientale* Linn., fam. *Pedaliaceae*) or good quality sesame cake by a process of expression or solvent extraction using solvent hexane conforming to IS 3470. The oil shall be refined by neutralization with alkali, bleached with bleaching earth or activated carbon or both, and deodourised with steam. The oil shall be practically odourless or have a very mild odour characteristics of sesame oil.

**4.1.2 *Type 2*** — The material shall be sesame oil obtained from clean and sound sesame seeds (*Sesamum indicum* Linn., syn. *Sesamum orientale* Linn., fam. *Pedaliaceae*) by a process of expression. The oil shall have the characteristics odour of sesame oil.

**4.2** The sesame oil shall be clear and free from rancidity, adulterants, sediment, suspend and other foreign matter, separated water and added colouring and flavouring substances.

**4.3 Admixture with Other Oils** — The sesame oil shall be free from admixture with other oils or adulterants, when tested according to the methods prescribed in IS 548 (Part 2).

**4.4** The sesame oil shall not contain Total Aflatoxins, more than 25 µg/kg, when tested by the method prescribed in 16287 : 2015/ISO 16050 : 2003- (Foodstuffs-Determination of aflatoxin B<sub>1</sub>, and the total content of aflatoxins B<sub>1</sub>, B<sub>2</sub>, G<sub>1</sub> and G<sub>2</sub> in cereals, nuts and derived products. The test for Total Aflatoxin shall be performed if agreed to between the purchaser and the supplier.

**4.5** The sesame oil shall also comply with the requirements given in Table 1, when tested as prescribed 5 of Table 1.

**Table 1 Requirements for Sesame Oil for Cosmetic Industry**  
( *Clauses 4.5 and 7.1* )

SI No.	Characteristic	Requirements		Method of Test, Ref to
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Moisture and insoluble impurities, percent by mass, <i>Max</i>	0.10	0.20	5 and 6 of IS 548 (Part 1)
ii)	Colour in a 1/4" cell on the Lovibond scale, expressed as Y + 5R, not deeper than	2	10	13 of IS 548 (Part 1)
iii)	Refractive index at 40 °C	1.4645 to 1.4665		10 of IS 548 (Part 1)
iv)	Relative density at 30/30 °C	0.915 to 0.919		11 of IS 548 (Part 1)
v)	Saponification value	185 to 193		15 of IS 548 (Part 1)
vi)	Iodine value, (Wij's)	103 to 115		14 of IS 548 (Part 1)
vii)	Acid value, <i>Max</i>	0.5	4.0	7 of IS 548 (Part 1)
viii)	Unsaponifiable matter, percent by mass, <i>Max</i>	1.5	1.5	8 of IS 548 (Part 1)
ix)	Bellier test (turbidity emperature), °C, <i>Max</i>	22.0	22.0	13 of IS 548 (Part 2)
x)	Flash point, Pensky-Martens (closed), °C, <i>Min</i>	250	—	IS 1448 (Part 21)
xi)	Test for rancidity	Shall be free from rancidity		Annex A
xii)	Peroxide value, meq/kg, <i>Max</i>	5.0	10.0	Annex B
xiii)	Hexane, ppm, <i>Max</i>	5.00	—	Annex C

## 5 PACKING AND MARKING

**5.1** The material shall be supplied in suitable well closed containers which do not deteriorate the product in quantity as agreed to between the purchaser and the supplier.

**5.2** The packages shall be securely closed and legibly marked with the following information:

- Name and type of the material;
- Manufacturer's name and/or his recognized trade-mark, if any;
- Net quantity of sesame oil in the container;
- Batch number, month and year of manufacture;
- Caution 'NOT FOR DIRECT EDIBLE CONSUMPTION' (either printed on the label affixed to the container or lithographed or stenciled thereon with indelible ink) in a type size of not less than 50 mm to be marked; and
- Any other information required by statutory authorities.

### 5.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity

assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

## 6 SAMPLING

**6.1** Representative samples of the sesame oil shall be drawn as prescribed under 3 of IS 548 (Part 1).

**6.2** Tests for all the requirements shall be carried out on a composite sample.

**6.3** The material shall be taken to have conformed to this standard if the composite sample passes all the tests. The test for Total Aflatoxin is an optional test and shall be performed if agreed to between the purchaser and the supplier.

## 7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water [*see* IS 1070 : 1992 Reagent grade water — Specification (*third revision*)] shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

## ANNEX A

[ Table 1, Item (xi) ]

## TEST FOR RANCIDITY

## A-1 REAGENTS

**A-1.1 Phloroglucinol Solution** — Dissolve 0.1 g of phloroglucinol in 100 ml of diethyl ether.

## A-2 PROCEDURE

Shake 10 ml of the material, melt if necessary, with 10 ml of concentrated hydrochloric acid and 10 ml of phloroglucinol solution. Shake for 1 minute.

**A-2.1** The material shall be taken to have passed the test if no pink colour develops.

## ANNEX B

[ Table 1, Sl No. (xii) ]

## DETERMINATION OF PEROXIDE VALUE

## B-1 REAGENTS

**B-1.1 Glacial Acetic Acid**

**B-1.2 Chloroform**

**B-1.3 Saturated Potassium Iodide Solution**

**B-1.4 Sodium Thiosulphate** — 0.01 N, accurately standardized.

Add 0.5 ml of starch solution, continue the titration, shaking vigorously until the blue colour just disappears (*A*). Carry out a blank determination under the same condition without adding any sample (*B*). The volume of 0.01 N sodium thiosulphate in the blank determination must not exceed 0.1 ml.

## B-2 PROCEDURE

Weigh  $5.0 \pm 0.5$  g of the sample in a 250 ml glass-stoppered conical flask. Add 30 ml of a mixture of 3 volumes glacial acetic acid and 2 volumes of chloroform. Swirl until dissolved, and add 0.5 ml of saturated potassium iodide solution. Allow to stand for exactly 1 min, with occasional shaking, add 30 ml of water. Titrate gradually, with continuous and vigorous shaking, with 0.01 N sodium thiosulphate solution until the yellow colour almost disappears.

## B-3 CALCULATION

Calculate the peroxide value from the expression:

$$\text{Peroxide value (meq/kg)} = \frac{(A - B) \times N}{M} \times 1000$$

where

*A* = volume in ml, of sodium thiosulphate solution required for titration;

*B* = volume in ml, of sodium thiosulphate solution required for blank titration;

*N* = normality of sodium thiosulphate solution; and

*M* = mass in g, of the sample.

## ANNEX C

[ Table 1, Sl No. (xiii) ]

## DETERMINATION OF HEXANE RESIDUES IN OILS AND FATS

## C-1 PRINCIPLE

The residual hexane content is the quantity of volatile hydrocarbons remaining in the fats and oils following processing involving the use of solvents. The volatile hydrocarbons are desorbed by heating the sample at

80 °C in a closed vessel after addition of an internal standard. After determination of a calibration factor, hydrocarbons in the head space are determined by gas chromatography using packed or capillary columns. Results are expressed as hexane,

in mg/kg (or ppm). The method is applicable to the determination of 'free' volatile hydrocarbons expressed in terms of hexane remaining in animal and vegetable fats and oils after extraction with hydrocarbon based solvents.

## C-2 APPARATUS

**C-2.1 Gas Chromatograph** — Gas chromatograph having:

- thermostatic column capable of maintaining the desired column temperature within  $\pm 1$  °C;
- sample inlet system, separately thermostated which can be maintained at a minimum temperature of 100 °C. If a capillary column is used, the inlet system must be capable of a 1/100 split injection. For serial analysis a headspace gas chromatograph with automatic sample injection and tempering bath is satisfactory; and
- flame ionization detector which can be separately thermostated and maintained at a minimum of 100 °C.

**C-2.2 Recorder** — If a recorder trace is to be used for calculating the composition of the samples analyzed, an electronic recorder of high precision is required or else use electronic integrator (*see C-2.3*).

**C-2.3 Electronic Integrator**, which permits rapid and accurate calculations.

**C-2.4 Chromatographic Column**, either packed or capillary column with the following minimum requirements:

- Packed column* — Stainless steel or glass, approximately 2 m long and 3.175 mm internal diameter with acid washed and silanized diatomaceous earth, 150-180 mm particle size (80-100 mesh chromosorb WAW is suitable), stationary phase — squalene consisting of 10 percent of packing.
- Capillary column* — Glass or fused silica approximately 30 m long and 0.3 mm internal diameter.
- Stationery phase* — Methyl polysiloxane (film thickness 0.2  $\mu$ m).

**C-2.5 Syringe** — 1 ml, 10 ml, 1000 ml capacity, gas tight.

**C-2.6 Septum Vial** — 20 ml capacity.

**C-2.7 Septa and Aluminium Caps Suitable for Septum Vials Together with Crimping Pliers** — The septa must be resistant to oils and solvents (butyl rubber or red rubber is recommended).

**C-2.8 Tongs**, suitable for holding septum vials.

**C-2.9 Heating Bath**, with clamps for holding septum vials, thermostatically regulated and capable of maintaining a temperature of 80 °C. For continuous operation glycerol is recommended as heating liquid.

## C-2.10 Shaking Machine

## C-3 REAGENTS

### C-3.1 Gases

- Carrier* — Helium (preferred for better resolution) or Nitrogen 99.99 percent pure, dried and containing a maximum of 10 mg O<sub>2</sub>/kg.
- Flame Ionization Detector* — Hydrogen, minimum purity 99.95 percent, air or oxygen, dry, hydrocarbon free (less than 2 ppm hydrocarbon equivalent to CH<sub>4</sub>).

**C-3.2 Technical Hexane or Light Petroleum**, with a composition similar to that used in industrial extraction or failing these *n*-hexane. For calibration, technical extraction hexane is preferred.

**C-3.3 *n*-Heptane (Internal standard)**, analytical reagent grade.

**C-3.4 Vegetable Oil**, solvent free, freshly refined and deodorized. The oil is to be used for calibration and should be of a similar nature as the sample. It should be free from extraction solvent (less than 0.01 percent).

## C-4 SAMPLING AND SAMPLE STORAGE

It is essential that loss of solvent from the sample be prevented. The laboratory sample should be in a completely sealed condition and stored at 4 °C. Plastic containers should not be used. Sample analysis should be carried out immediately when the sample container is opened.

## C-5 GC OPERATING CONDITIONS

Carrier gas flow depends on the carrier gas and the type of column being used for analysis and should be optimized accordingly. The flow of hydrogen and air or oxygen to the FID should be optimized according to the manufacturer's recommendation. Injector and detector temperatures should be set at about 120 °C. The column should be maintained at 40 °C.

## C-6 PROCEDURE

### C-6.1 Determination of the Calibration Factor

Weigh to the nearest 0.01 g, 5 g of solvent free vegetable oil (*see C-3.4*) into each of the 7 septum vials. Seal each vial with a septum and cap. By means of a syringe add technical hexane to 6 of the seven vials (in the vial with no added solvent is the blank) according to the following table:

ml/5 g	0.5	1	2	4	7	10
mg/100 g	67	134	268	536	938	1340

One vial remains without the addition of solvent.

If *n*-hexane is used for calibration the following table applies:

ml/5 g	0.5	1	2	4	7	10
mg/100 g	66	132	264	528	924	1320

Shake the 6 vials containing the solvent in the shaking machine vigorously for 1 h. Using the syringe add 5 ml of internal standard (*see C-3.3*) to each of the 7 vials. Successively immerse the vials up to the neck in the heating bath at 80°C at intervals of approximately 15 min. This time interval depends on the duration of the GC analysis which is complete on the elution of the internal standard (*n*-heptane). The samples must be placed in the heating at intervals such that each sample is tempered for exactly 60 min.

Warm the gas tight syringe to 60 °C. After tempering at 80°C for exactly 60 min and without removing the vial from the heating bath, use the gas tight syringe and withdraw through the septum 1 000 µl (1 ml) of the head space above the oil. Inject immediately into the gas chromatograph. For each of the vial containing added solvent a calibration factor *F* may be determined by the following formula:

$$F = \frac{C_s \times A_i}{(A_H - A_B - A_i) \times C_1}$$

where

$A_H$  = total peak area of solvent hydrocarbons including the area of internal standard present in the spiked oil<sup>1</sup>.

$A_B$  = peak area of the solvent hydrocarbons present in the oil to which solvent has not been added (blank) less the peak area of the internal standard.

$A_i$  = peak area corresponding to the internal standard in the spiked samples.

$C_1$  = quantity of the internal standard added expressed, in mg/kg, of the oil.

$C_s$  = quantity of technical hexane added to the oil present in the vial expressed, in mg/kg, of the oil.

NOTE<sup>1</sup> — For identification purposes a typical chromatogram of solvent composition should be obtained. Hydrocarbons which usually make up the technical hexane are 2 methyl pentane, 3 methyl pentane, methyl cyclopentane, cyclohexane, etc. Do not include peaks due to oxidation products which may be present in significant amounts.

Express the results to the third decimal place.

Calibration factors of the six standards should be approximately the same. The mean calibration factor should be 0.45, if *n*-heptane is used and 0.57, if cyclohexane is used.

The factor (*F*) so evaluated can be used for determining vial quantities of hexane less than 60 mg/kg. If the value of *F* found for the vial containing 0.5 ml of hexane is significantly below the mean value, this deviation is probably due to difficulty in introducing exactly 0.5 ml and this determination must be either eliminated or repeated. For quantities of hexane between 10 and 20 mg/kg it is better to prepare calibration standards by adding 2 ml of internal standard instead of 0.5 ml.

### C-6.2 Sample Analysis

Weigh to the nearest 0.01 g, 5 g of the test sample into a septum vial as quickly as possible and close immediately with a septum and cap. Using a syringe add through the septum exactly 5ml of the internal standard. Shake vigorously by hand for about 1 min and then immerse the vial up to the neck in the heating bath. At 80 °C for exactly 60 min. Warm the gas tight syringe to 60 °C. After tempering at 80 °C for exactly 60 min use the gas tight syringe and take from the vial without removing it from the bath 1 000 µl (1 ml) of the head space above the sample. Immediately inject into the gas chromatograph. Carry out two determinations in rapid succession on each sample.

### C-7 CALCULATION

The residual solvent expressed, in mg/kg (ppm), is given by the following formula:

$$W = \frac{(A_H - A_i) \times F \times C_1}{A_i}$$

where

$A_H$  = total peak area of solvent hydrocarbons<sup>1</sup> including the area of internal standard;

$A_i$  = peak area corresponding to internal standard in the sample;

$C_1$  = quantity of the internal standard added<sup>2</sup>, in mg/kg; and

$F$  = calibration factor obtained in procedure.

#### NOTES

<sup>1</sup> Hydrocarbons which usually make up the technical solvents are 2-methyl pentane, 3-methyl pentane, methyl cyclopentane, cyclohexane, etc. Do not include peaks due to the oxidation products. Some of these products may be present in significant amount.

<sup>2</sup> For an addition of 5 ml of heptane/5 g of sample  $C_1$  = 680 mg/kg and  $C_1$  = 750 mg/kg, if cyclohexane is used.

Report as the final result the mean of the results of two determinations.



## ANNEX K

( Foreword )

## COMMITTEE COMPOSITION

Cosmetics Sectional Committee, PCD 19

<i>Organization</i>	<i>Representative(s)</i>
Drugs Controller General (INDIA), Delhi	DR V. G. SOMANI ( <b>Chairman</b> )
All India Cosmetic Manufacturers Association, Mumbai	MS KAJAL ANAND DR VIRENDRA V. CHAVAN ( <i>Alternate</i> )
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Cavinkare Private Limited, Chennai	DR T. KUMAR DR GIREESH KUMAR ( <i>Alternate I</i> ) DR S. SANKAR KALIDAS ( <i>Alternate II</i> )
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Central Drugs Testing Laboratory (CDTL), Chennai	MS C. VIJAYA LAKSHMI DR J. UMA MAHESWARI ( <i>Alternate</i> )
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Drugs Control Department, Delhi	SHRI A. K. NASA SHRI K. R. CHAWLA ( <i>Alternate</i> )
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Essential Oil Association of India (EOAI), Noida	SHRI AJAY K. JAIN DR ANITA BHATNAGAR JAIN SHRI DINESH KUMAR TIWARI ( <i>Alternate</i> )
Food Safety and Drug Administration, Lucknow	DR H. G. KOSHIA SHRI V. R. SHAH ( <i>Alternate</i> )
Food and Drugs Control Administration Gujarat, Gandhinagar	SHRI NARENDER KUMAR AHOOJA SHRI MANMOHAN TANEJA ( <i>Alternate</i> )
Food and Drugs Administration Haryana, Panchkula	SHRI O. S. SADHWANI
Food and Drugs Administration Maharashtra, Mumbai	SHRI HASMUKH PATEL
Fragrance and Flavours Association of India, (FAFAI), Mumbai	



<i>Organization</i>	<i>Representative(s)</i>
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Godrej Consumers Products Limited, Mumbai	MS RUPINDER KAUR RAWAT DR MANOJ GAUR ( <i>Alternate</i> )
Hindustan Lever Limited (HUL), Mumbai	MS VRINDA RAJWADE
Hygienic Research Institute Private Limited, Mumbai	DR JAYASHREE ANAND SHRI MANOJ SARKAR ( <i>Alternate</i> )
Indian Pharmacopoeia Commission (IPC), Ghaziabad	DR ANIL KR TEOTIA DR MANOJ KR PANDEY ( <i>Alternate</i> )
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Kelkar Education Trusts (KETS) Scientific Research Centre, Mumbai	DR S. S. BARVE
Loreal India Private Limited, Mumbai	MS VEENA BALGI MS RUPALI TURAKHIYA ( <i>Alternate</i> )
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Ministry of AYUSH, Delhi	DR D. C. KATOCH
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PETA India, Mumbai	SHRI MANILAL VALLIYATE MS DIPTI M. KAPOOR
The Himalaya Drug Company, Bengaluru	DR SUNDARAM RAMACHANDRAN DR KRISHNAN SRIRAMAN ( <i>Alternate</i> )
Voluntary Organization In Interest of Consumer Education (VOICE), Delhi	DR M. A.U. KHAN
Bureau of Indian Standards, Jammu	SHRIMATI NISHA BURA
BIS Directorate General	SHRIMATI NAGAMANI T., SCIENTIST 'E' AND HEAD (PCD) [REPRESENTING DIRECTOR GENERAL ( <i>Ex-officio</i> )]

*Member Secretary*

SHRIMATI D. UMA  
SCIENTIST 'D' (PCD), BIS

## Composition of Raw materials Subcommittee, PCD 19: 1

<i>Organization</i>	<i>Representative(s)</i>
The Himalaya Drug Company, Bengaluru	DR SUNDARAM RAMACHANDRAN ( <b>Convener</b> )
BASF India Limited, Mumbai	SHRI UDAY KULKARNI SHRI VIPUL BHATT ( <i>Alternate</i> )
Central Drug Testing Laboratory, Kolkata	SHRI HARIHARAN
Central Drugs Testing Laboratory, Chennai	SHRIMATI C. VIJAYA LAKSHMI DR J. UMA MAHESWARI ( <i>Alternate</i> )
Chemstar Limited, Thane	SHRI SUNIL JOSHI
Consumer Education and Research Centre, Ahmedabad	DR C. J. SHISHOO SHRI H. S. TRIPATHI ( <i>Alternate</i> )
Dabur India Limited, Sahibabad	DR S. K. LUTHRA SHRI PRASUN BANDYOPADHYAY ( <i>Alternate I</i> ) SHRI SHIVAJI RAI ( <i>Alternate II</i> )
Drugs Testing Laboratory, Thiruvananthapuram	SHRI SUTHA T. SHRI LINEX D. SILVA ( <i>Alternate</i> )
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Food and Drugs Laboratory, Vadodara	SHRI H. B. CHAUDHARI SHRI K. N. PATEL ( <i>Alternate</i> )
Galaxy Surfactants Limited, Mumbai	SHRI R. K. SINGH SHRI SAGAR TRAILOKYA ( <i>Alternate I</i> ) SHRI PRAMOD SABAT ( <i>Alternate II</i> )
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Sudarshan Chemical Industries Limited, Pune	SHRI S. H. HARSULE SHRI M. D. METTELLOO ( <i>Alternate</i> )



## Bureau of Indian Standards

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### Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 19 (13239).

### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

## BUREAU OF INDIAN STANDARDS

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